We claim:

A process for the preparation of 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic esters of the formula I,

I

in which the substituents, independently of one another, have the following meanings:

 $R^1$  and  $R^2$ 

are  $C_1-C_6$ -alkyl,  $C_3-C_{10}$ -cycloalkyl;

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 $R^3$  is  $C_1-C_{12}$ -alkyl,  $C_3-C_{10}$ -cycloalkyl

by

25 I. reaction of 3-N,N-dialkylaminophenol of the formula II, in which R<sup>1</sup> and R<sup>2</sup> have the meanings given above, with phthalic anhydride of the formula III to give 2-(4-N,N-dialkylamino-2-hydroxybenzoyl)benzoic acid of the formula IV and

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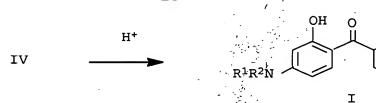
II. subsequent esterification of the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic acid of the formula IV formed in stage I with a  $C_1$ - $C_{12}$ -alcohol or a cyclic  $C_3$ - $C_{10}$ -alcohol in the presence of an acidic catalyst to give the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic ester of the formula I,

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COOR3

which comprises purifying the ester of the formula I formed in a further process stage III by treatment with an adsorbent and/or by distillation.

2. A process as claimed in claim'1, wherein the adsorbent is a substance chosen from the group consisting of activated carbons, aluminum oxides, zeolites and silica gels.

15 3. A process as claimed in claim 1 or 2, wherein the esterification in the process stage II is carried out in the presence of sulfuric acid as catalyst.

- 4. A process as claimed in any of claims 1 to 3, wherein the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl)benzoic ester of the formula I formed is crystallized prior to the treatment with an adsorbent and/or distillation.
- 5. A process as claimed in any of claims 1 to 4, wherein the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl)benzoic ester of the formula I formed comprises less than 10 ppm of rhodamine.
- A process as claimed in any of claims 1 to 5, wherein the benzoic ester is n-hexyl 2-(4-N,N-diethylamino-2-hydroxybenzoyl) benzoate of the formula Ia

7. A process as claimed in any of claims 1 to 6, wherein, in the process stage III, the adsorbent used is activated carbon or silica gel.

Ιa

8. A process as claimed in claim 7, wherein, in process stage

III, the ester is purified by treatment with activated carbon and subsequent distillation.

- 9. A process as claimed in claim 8, wherein, in the process stage III
- a. the ester is dissolved in a nonpolar solvent at a temperature in the range from 10°C to 100°C,
  - b. this solution is passed over a granular activated carbon bed at a temperature in the range from 20°C to 100°C,
- 10 c. the ester, after passing through the granular activated carbon bed, is separated off from the solvent by distillation.
- 10. A process as claimed in claim 9, wherein the solvent used in the process step IIIa is cyclohexane or toluene.
  - 11. A process for the preparation of n-hexyl 2-(4-N,N-diethylamino-2-hydroxybenzoyl)benzoate of the formula Ia

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by

30 I. reaction of 3-N,N-diethylaminophenol of the formula IIa with phthalic anhydride of the formula III to give 2-(4-N,N-diethylamino-2-hydroxybenzoyl)benzoic acid of the formula IVa,

Ιa

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$$(CH_3CH_2)_2N \longrightarrow (CH_3CH_2)_2N \longrightarrow IVa$$

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II. esterification of the 2-(4-N,N-diethylamino-2hydroxybenzoyl)benzoic acid of the formula IVa formed in stage I in hexanol in the presence of sulfuric acid to give n-hexyl 5

2-(4-N,N-diethylamino-2-hydroxy] benzoyl) benzoate of the formula Ia

IVa hexanol/H+ (CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>N OH O COO-n-hexyl

Ιa

and isolation of the n-hexyl ester Ia in crystalline form,

III.

- a. dissolution of the n-hexyl ester Ia in toluene or hexanol at a temperature in the range from 25°C to 50°C,
  - b. metering of this solution over a granular activated carbon bed or a silica gel bed at a temperature in the range from 25°C to 50°C and
    - c. subsequent isolation of the n-hexyl ester by separating off the toluene and/or hexanol by distillation.

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Preparation of 2-(4-N,N-dialkylamino-2-hydroxybenzoyl)benzoic esters

## 5 Abstract

The invention relates to a process for the preparation of 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic esters of the formula I,

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$$\begin{array}{c|c} \text{OH} & \text{O} & \text{COOR}^3 \\ \hline \\ R^1R^2N & & & \end{array}$$

in which the substituents  $R^1$  to  $R^3$ , independently of one another, have the meanings given in the description by:

I. reaction of 3-N,N-dialkylaminophenol of the formula II with phthalic anhydride of the formula III to give 2-(4-N,N-dialkylamino-2-hydroxybenzoyl)benzoic acid of the formula IV and

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30 II. subsequent esterification of the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic acid of the formula IV formed in stage I with a  $C_1$ - $C_{12}$ -alcohol or a cyclic  $C_3$ - $C_{10}$ -alcohol in the presence of an acidic catalyst to give the 2-(4-N,N-dialkylamino-2-hydroxybenzoyl) benzoic ester of the formula I,

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IV 
$$\mathbb{R}^{1}\mathbb{R}^{2}\mathbb{N}$$
  $\mathbb{I}$ 

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which comprises purifying the ester of the formula I formed in a further process stage III by treatment with an adsorbent and/or by distillation.

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